

Remarks

Reconsideration and approval of this application are respectfully requested.

No claims are added or cancelled by this amendment. Claims 1-21 thus remain under consideration in this application.

Claim 10 is revised merely to provide antecedent basis for the term "alkaline salt." It is believed that no new matter is added by this amendment.

Entry of this amendment after final action is appropriate because it makes no substantive change and will bring the application into condition for immediate allowance. No additional searching should be required because the amendment adds no new limitations to the claims.

Applicant acknowledges, with thanks, that the previously stated rejections under 35 U.S.C. 112 have been withdrawn.

All the pending claims continue to stand rejected under 35 U.S.C. 103(a) from some combination of publications including Ruff et al (5,066,472) or Breneman (4,743,344) in view of WO 03/033115 (using Hirano et al, US 2004/0258596 as unofficial English translation) and Terry et al (3,900,312) and Keller et al (3,878,291).

In the response filed on April 23, 2010, the applicant asserted that all the prior art rejections should be withdrawn because WO 03/033115 is essential to all the stated rejections but is not prior art.

In the Office action of July 13, 2010, the examiner asserted that WO 03/033115 is prior art because the priority application (U.S. Provisional Application No. 60/459,867, filed April 1, 2003) allegedly does not support certain limitations found in the pending claims.

Reconsideration of this issue and withdrawal of the citation of WO 03/033115 are requested.

With regard to claim 1 (and in part with regard to claims 8 and 15), the Office action mentions three limitations that allegedly are not supported by the disclosure of Provisional Application No. 60/459,867.

1. "low volatility"

The phrase "lower volatility components including at least one metal chloride" appears in claims 1 and 7 of Provisional Application No. 60/459,867 (Exhibit A). (The claims originally filed in an application are a portion of the disclosure of that application. In particular, the "specification" of a patent application includes the "claims.") Thus there is clear support in the priority application, filed on April 1, 2003, for the term "low volatility."

2. "in the absence of liquid water"

The text of Provisional Application No. 60/459,867 provides clear support for the term "in the absence of liquid water."

Specifically, at page 9, lines 15-22, Provisional Application No. 60/459,867 (Exhibit B) states:

"Because the moisture is carried into the process in the form of a hydrated solid, **there is substantially no free moisture within the process.** The metal chlorides, for example aluminum chloride, have a much stronger affinity for moisture than the alkali carbonate hydrate. Thus **the environment within the mixer/drier is maintained in a state where no condensation of water or hydrochloric acid occurs,** thus reducing the corrosive effect. The mixer/drier can be constructed of a duplex stainless steel alloy such as Ferillium that is much less expensive than the nickel/chromium/molybdenum alloys or glass enameled equipment that would otherwise be required." (Emphasis added)

Provisional Application No. 60/459,867 makes it clear that avoidance of liquid water / HCl is a significant benefit of the claimed methods. The problems of liquid water are explained, for example, in the background of Provisional Application No. 60/459,867, at page 3, lines 7-15 (Exhibit C):

"All of these present processes, without exception, result in contacting the residue with liquid water. **The reaction of water with either the residual chlorosilanes or the metal chloride impurities results in the formation of corrosive hydrochloric acid.** Therefore, the process equipment must be constructed of corrosion resistant materials. Leaks and spills provide a high likelihood of environmental contamination and worker exposure to corrosive materials. Furthermore, the aqueous hydrolysis of chlorosilanes results in the formation of

silica solids not only within the reaction mixture, but the solids can deposit on the interior portions of the equipment causing a process limiting build-up or plugging of pipelines.” (Emphasis added)

Thus there is clear support in the priority application, filed on April 1, 2003, for the term “in the absence of liquid water.”

3. “at a temperature greater than 80°C”

The Office action observes:

“It is noted that in the Provisional Application '867, it is disclosed in step 3) ‘Subjecting the substantially non-volatile solid residues to the action of selected alkaline hydrate solids at a temperature in excess of about 80°C to yield a stable substantially neutral’ . . .”

The Office action then goes on to state:

“however, the temperature in this step is only for a process treating the residue from a chlorosilane manufacturing process, not for a process treating a generic ‘flowable solid material’ as required in Applicant’s claim 1. Also this temperature is only applicable when ‘selected alkaline hydrate solids’ are used, not for a generic ‘hydrate’ as now required in Applicants’ claims 8 and 15.”

Applicant respectfully asserts that such conclusions are not correct.

Claim 13 of Provisional Application No. 60/459,867 (Exhibit D) called for:

“A method of processing a **flowable solid material that includes at least one metal chloride**, the method comprising: contacting a flowable solid material that includes at least one metal chloride with a **flowable solid hydrate** in a vessel at a **temperature from 80° to 150°C** such that the at least one metal chloride reacts with the hydrate . . .” (Emphasis added)

It is thus clear that the priority application, filed on April 1, 2003, contemplated 80°C as the base temperature for treating a “flowable solid material that includes at least one metal chloride” in contact with “a flowable solid hydrate.” The disclosure of Provisional Application No. 60/459,867 clearly was not limited just to “a process treating the residue from a chlorosilane manufacturing process” or to “selected alkaline hydrate solids.”

Conclusion

The rejections of claims 1, 8, and 15 under 35 U.S.C. 103(a) should be withdrawn because WO 03/033115 is essential to all the stated rejections, but is not prior art.

Claims 1, 8, and 15 are fully supported by Provisional Application No. 60/459,867 and thus have an effective filing date of April 1, 2003.

That filing date is before the date of publication of WO 03/033115 (in Japanese language). Therefore, WO 03/033115 is not prior art with regard to claims 1, 8, and 15.

Claims 2-7, 9-14, and 16-21 depend from claims 1, 8, and 15. Claims 2-7, 9-14, and 16-21 thus are allowable for at least the reason that they depend from allowable claims.

However, each of claims 2-7, 9-14, and 16-21 defines a further improvement in its own right so that each such claim should be considered separately.

Reconsideration and issuance of a Notice of Allowance are requested.

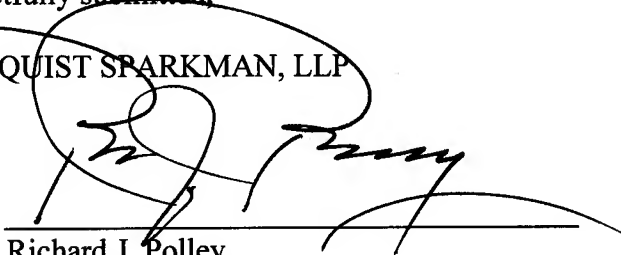
Should any issues remain to be discussed, the Examiner is urged to call the undersigned for the purpose of a telephone interview.

Respectfully submitted,

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Claims:

1. A method of processing the residue from a chlorosilane manufacturing process, the method comprising:
 - concentrating a residue mixture containing volatile chlorosilanes and lower
5 volatility components including at least one metal chloride in a drier unit suitable for processing a solid fraction;
separating volatile chlorosilane vapors from the mixture for recycle or reprocessing;
contacting the remaining substantially chlorosilane-free solid residue with a
10 hydrate at a temperature from 80° to 150°C such that the at least one metal chloride reacts with the hydrate; and
discharging the resulting flowable mixture for disposal or metals recovery.
2. The method of claim 1 further comprising contacting the substantially
15 chlorosilane-free solid residue with an alkaline salt to increase the pH of the resulting flowable mixture.
3. The method of claim 2 further comprising simultaneously contacting the remaining substantially chlorosilane-free solid residue with a hydrate and contacting the
20 remaining substantially chlorosilane-free solid residue with the alkaline salt.
4. The method of claim 3 wherein the contacting of the remaining substantially chlorosilane-free solid residue with a hydrate and the contacting of the remaining substantially chlorosilane-free solid residue with the alkaline salt is
25 accomplished by contacting of the remaining substantially chlorosilane-free solid residue with mechanically refined trona, which is a natural form of sodium sesquicarbonate, is a hydrated mineral, and provides an alkaline salt.

Exhibit A (page 1/2)

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5. The method of claim 2 wherein:
the alkaline salt is calcium carbonate; and
the hydrated mineral is damp natural soil.

5 6. The method of claim 2 wherein:
the alkaline salt is magnesium hydroxide; and
the hydrated mineral is montmorillonite clay.

7. A method of processing the residue from a chlorosilane manufacturing
10 process, the method comprising:
concentrating a residue mixture containing volatile chlorosilanes and lower
volatility components including at least one metal chloride in a drier unit suitable for
processing a solid fraction in the presence of finely milled sodium chloride;
separating volatile chlorosilane vapors from the mixture for recycle or
15 reprocessing;
contacting the remaining substantially chlorosilane-free solid residue with a
hydrate at a temperature from 80° to 150°C such that the at least one metal chloride
reacts with the hydrate; and
discharging the resulting flowable mixture for disposal or metals recovery.

20 8. The method of claim 7 further comprising contacting the substantially
chlorosilane-free solid residue with an alkaline salt to increase the pH of the resulting
flowable mixture.

25 9. The method of claim 8 further comprising simultaneously contacting the
remaining substantially chlorosilane-free solid residue with a hydrate and contacting the
remaining substantially chlorosilane-free solid residue with the alkaline salt.

Exhibit A (page 2/2)

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example, may or may not be alkaline depending on the where they are acquired, and thus might or might not benefit from the use of a separate alkaline salt. Calcium carbonate and magnesium hydroxide are examples of separate alkaline salts that could be added to neutralize HCl. Such materials are best introduced as flowable solids, such as powder or
5 granules. If an excess of a salt is provided, the resulting mixture will be somewhat basic.

The resulting neutralized residue also is in the form of a flowable solid material, typically powder or granules, and can be safely disposed of in an environmentally acceptable manner. After discharge of the neutralized solids, the mixer/drier is ready for a subsequent charge of chlorosilane slurry with out need for further clean-up.

10 The discharged solids meet the requirements for non-hazardous solid waste by the "TCLP" or Toxic Characteristic Leaching Protocol⁵. Alternatively, if valuable metals, such as copper, nickel or silver and the like, are used as catalysts or promoters in the production of chlorosilanes, the neutralized flowable residue solids can be made available for recovery of those metals by conventional hydrometallurgy extraction techniques.

15 Because the moisture is carried into the process in the form of a hydrated solid, there is substantially no free moisture within the process. The metal chlorides, for example aluminum chloride, have a much stronger affinity for moisture than the alkali carbonate hydrate. Thus the environment within the mixer/drier is maintained in a state where no condensation of water or hydrochloric acid occurs, thus reducing the corrosive effect. The
20 mixer/drier can be constructed of a duplex stainless steel alloy such as Ferillium that is much less expensive than the nickel/chromium/molybdenum alloys or glass enameled equipment that would otherwise be required.

Alternatively, the solid may be sold to a hydrometallurgy extraction processor where the valuable metals could be selectively reclaimed.

⁵ 40 CFR 268.49

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Other means have been proposed to treat residues from the purification of chlorosilanes such as are generated during the production of polycrystalline silicon. Those processes involve hydrolysis of the residues, but neutralization of the resulting hydrochloric acid and filtration to remove the co-product silica both involve the use of expensive acid resistant equipment and the high maintenance costs associated with the processing of corrosive hydrochloric acid. Filtration of the resulting slurries is difficult and many times is just not possible as the hydrolysis reactions form gels and ultra-fine particles. All of these present processes, without exception, result in contacting the residue with liquid water. The reaction of water with either the residual chlorosilanes or the metal chloride impurities results in the formation of corrosive hydrochloric acid. Therefore, the process equipment must be constructed of corrosion resistant materials. Leaks and spills provide a high likelihood of environmental contamination and worker exposure to corrosive materials. Furthermore, the aqueous hydrolysis of chlorosilanes results in the formation of silica solids not only within the reaction mixture, but the solids can deposit on the interior portions of the equipment causing a process limiting build-up or plugging of pipelines.

Summary of the Disclosure

It would be useful to provide a method for economically processing the residues from chlorosilane production to yield a waste product that can be readily disposed of, and preferably, to completely recover valuable chlorosilanes. It also would be desirable for the process to afford an opportunity to reclaim valuable metals by well known extractive metallurgy techniques. It further would be useful to provide a process which can be conducted without need for equipment constructed of the exotic metals or materials required to be resistant to the corrosion of hydrochloric acid.

Described herein are processes by which such residues can be dried and the volatile chlorosilanes (or titanium chlorides) recovered for re-use while the non-volatile solids, containing reactive metal chlorides, are treated with an alkali carbonate or bicarbonate

Exhibit C

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10. The method of claim 9 wherein the contacting of the remaining substantially chlorosilane-free solid residue with a hydrate and the contacting of the remaining substantially chlorosilane-free solid residue with the alkaline salt is accomplished by contacting of the remaining substantially chlorosilane-free solid residue with mechanically refined trona, which is a natural form of sodium sesquicarbonate, is a hydrated mineral, and provides an alkaline salt.

11. The method of claim 8 wherein:
the alkaline salt is calcium carbonate; and
the hydrated mineral is damp natural soil.

12. The method of claim 8 wherein:
the alkaline salt is magnesium hydroxide; and
the hydrated mineral is montmorillonite clay.

13. A method of processing a flowable solid material that includes at least one metal chloride, the method comprising:
contacting a flowable solid material that includes at least one metal chloride with a flowable solid hydrate in a vessel at a temperature from 80° to 150°C such that the at least one metal chloride reacts with the hydrate; and
discharging the resulting flowable mixture of solids for disposal or metals recovery.

Exhibit D